[Geochemical Analysis & Lab]

EXPERIMENT 7: Silica Determination Using Molybdosilicate Method

**Caution**: Fully understand the whole procedures before getting started!!!

**Preparation**:

* Sample 50 mL
* HCl, NaOH, ammonium molybdate (NH4)6Mo7O24.4H2O), oxalic acid (H2C2O4.H2O), sodium metasilicate nonahydrtae (Na2SiO3.9H2O)
* UV-Visible photometer, sample cells (more than two)
* Chemical balance
* Polyethylene bottle; 250 mL (3), 1000 mL (1)
* Pipet; graduated 10 mL (1), 5 mL (1), 10 mL (1), 20 mL (1), 100 mL (1)
* Beaker; 500 mL (1), 250 mL (1), 100 mL (12)
* Volumetric flask; 1 L (1), 100 mL (3), 50 mL (10)
* Hot plate, water bath
* Glass rod
* Timer (any alarm clock)

**Procedures:**

1. Reagent Preparation
2. 1:1 HCl
   1. Using a 100mL volumetric pipette, add 100mL deionized water (DIW) into a clean 250mL PE bottle
   2. Wipe out al moistures outside the pipette, and use the same pipette to add 100mL c-HCl to the same PE bottle
   3. Close the cap and mark with date
3. Ammonium molybdate solution
   1. Add 100mL DIW into a clean 500mL beaker
   2. Place the 500mL beaker on a hot plate and heat not to boil
   3. Add 10g ammonium molybdate to another 100mL beaker
   4. Add 30mL DIW from hot 500mL beaker to the 100mL beaker and transfer the 100mL beaker to a water bath
   5. Slowly stir the solution with a glass rod until complete dissolution (if there are still residues, add 10mL DIW and stir again)
   6. Transfer the solution to a 100mL volumetric flask, rinse 3-4 times the beaker and add the rinsing water to the volumetric flask. Dilute to the mark.
   7. Store the solution in a clean PE bottle and mark.
4. SiO2 standard solutions
   1. Add 4.73g sodium metasilicate nonahydrate (SMN) into a clean 250mL beaker, Dissolve it with 100mL DIW.
   2. Transfer the solution into a 1L volumetric flask, rinse the beaker 3-4 times and add the rinsing water to the volumetric flask. Dilute to the mark.
   3. Store the solution in a clean 1L PE bottle, mark as “1000 mg/L SiO2 stock solution”. (The actual concentration should be calculated with the amount of SMN weighed in step a)
5. Determination of SiO2 in sample
6. Warming up the photometer – for one hour before the measurement
7. Coloring the sample and standard solutions
   1. Take 25mL of the 1000mg/L SiO2 stock solution and put into 500mL volumetric flask. Dilute to he mark (This is ca. 50mg/L solution)
   2. Take 1, 5, 10, 15, and 20mL of 50mg/L solution and transfer each to a 50mL volumetric flask. Dilute to the mark (Their concentrations are ca. 1, 5, 10, 15, and 20mg/L SiO2)
   3. Prepare 7 100mL beakers. Decant each standard solution to a separate beaker. One of the two other beakers will have50mL sample and the last one 50mL DIW. Mark the beakers appropriately
   4. Add 1mL 1:1 HCl and then 2mL ammonium molybdate solution to each beaker.
   5. Thoroughly mix the solutions and let them stand for 5 to 10 mins
   6. Add 2mL oxalic acid solution and then thoroughly mix again. Let them stand at least 2 mins.
   7. Set the wavelength of the photometer 410nm. Check if the cell is clean
   8. Measure the absorbance of the solutions. The whole experiments from step d to h should be done within 13 mins.
   9. Subtract the absorbance value of DIW from those of the sample and standards (This is going to be the corrected absorbance A’)
   10. Plot concentrations on the horizontal axis and A’ of the standards on the vertical axis. Do the linear regression and obtain an equation.
   11. Put the A’ value of the sample into the equation and obtain the SiO2 concentration (c) of the sample
   12. The corrected SiO2 concentration of the sample will be calculated with c’=c\*55/50 (mg/L).

**Notes:**